Fourier transform infrared spectroscopy approach for measurements of photoluminescence and electroluminescence in mid-infrared

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An improved Fourier transform infrared spectroscopy approach adapting to photoluminescence and electroluminescence measurements in mid-infrared has been developed, in which diode-pumped solid-state excitation lasers were adopted for photoluminescence excitation. In this approach, three different Fourier transform infrared modes of rapid scan, double modulation, and step scan were software switchable without changing the hardware or connections. The advantages and limitations of each mode were analyzed in detail. Using this approach a group of III–V and II–VI samples from near-infrared extending to mid-infrared with photoluminescence intensities in a wider range have been characterized at room temperature to demonstrate the validity and overall performances of the system. The weaker electroluminescence of quantum cascade lasers in mid-infrared band was also surveyed at different resolutions. Results show that for samples with relatively strong photoluminescence or electroluminescence overlapped with background, rapid scan mode is the most preferable. For weaker photoluminescence or electroluminescence overlapped with background, double modulation is the most effective mode. To get a better signal noise ratio when weaker photoluminescence or electroluminescence signal has been observed in double modulation mode, switching to step scan mode should be an advisable option despite the long data acquiring time and limited resolution.

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of the accessional system. In this work, with the comparative analysis of the system, an improved FTIR-PL approach adopting diode-pumped solid-state (DPSS) excitation lasers have been developed. Using this approach, a group of samples extending from NIR to MIR with quite different PL intensities have been characterized to demonstrate the system validity.

II. FTIR-PL CONSIDERATION AND CONFIGURATION

In FTIR spectrometer, the optical frequency \( v \) (wave numbers, \( \text{cm}^{-1} \)) is transformed to Fourier frequency of \( f_F = 2v \) (Hz) through a Michelson interferometer with moving mirror speed \( v \) (cm/s). In general, the moving mirror speed \( v \) could be changed 2–3 orders and scaled at wavelength of mirror speed of \( v \) (cm/s). In this approach, the modulation frequency of the excitation laser becomes difficult, the response of the system, and random interference from the circumstance become uncontrollable, the patience of the operator has also been tested. Furthermore, in SS mode, the “instant” response feature of the FTIR was lost, the operator just could not see the effects of operation instantly, such as in the optical path alignment but at least minutes later. Without the help of catching a weak traceable PL signal at other modes, the operation of SS mode independently becomes extremely difficult, especially for a new operator with an unknown sample at hand. These non-theoretical but practical demerits restrict the adoption of SS mode in FTIR-PL remarkably.

Based on above considerations, an improved FTIR PL and EL configuration adopting DPSS excitation laser was developed as shown schematically in Fig. 1. With the progress of high power semiconductor lasers, DPSS laser is becoming popular for its high reliability, moderate stability, small size, and lower price. Since this type of solid-state laser is pumped by electrically driven semiconductor diode laser, it could be electrically modulated inherently. The DPSS lasers are also inherently free from the strong non-laser plasma emission.
FIG. 1. Schematic diagram of the FTIR spectroscopy setup for PL and EL.

(quiet troublesome in PL measurements) of gas lasers. For most DPSS laser products, analog or digital modulation input port of 0–5 V scale has become a standard option, the modulation frequency could be from dc to tens of kHz, just fit the needs. The DPSS lasers have many wavelength options, with different power efficiencies and so on. For numerous wavelengths, the 808 nm diode laser pumped 1064 nm DPSS laser and its frequency doubled 532 nm DPSS laser are preferred for their high power efficiencies and therefore lower price for unit power. The power efficiency could exceed 50% for 1064 nm, and attains 20%–35% for 532 nm DPSS lasers. Normally, the spectral feature and beam quality of low-end DPSS laser products are good enough for PL excitations already. Visible 532 nm DPSS laser could be a direct substitute of the 514.5 nm argon laser frequently used in previous PL system. For reaching deeper excitation depth and higher excitation power, the infrared 1064 nm DPSS laser should be a good choice. Although it is invisible to human eyes, it still could be clearly seen by Si CCD or CMOS image sensors, so the adjustment of the system is still quite easy. Certainly the diode laser itself could also be used for PL excitation more directly, but its beam quality remains a problem, further shaping of the beam is troublesome.

As shown in Fig. 1, our system was based on a Nicolet Magna-IR System 860 FTIR spectrometer (a quite old version) with simultaneous synchronous technique module. The FTIR accessory for PL and EL was built on a 30 × 30 cm² attachable optical breadboard with beam height match to the spectrometer. The excitation light from the exchangeable DPSS laser head (532 nm or 1064 nm with the same size) was steered by an adjustable reflecting mirror, then focused on the sample by a small focus lens (with focus length of a few centimeters). The sample was put on an adjustable sample holder X-Y stage horizontally. The beam diameter of the DPSS lasers was normally <3 mm, so the spot diameter through the focus lens on the sample could be below 1 mm. The laser beam reflected from the sample was blocked and absorbed using a covering. The excited PL from the sample were collected and collimated towards the emission port of FTIR spectrometer using a 90° off-axis parabolic mirror (OAPM), its diameter was 2 in. and focus length was 3 in. The OAPM is unselective to wavelength, so this FTIR-PL accessory was suitable from visible to far-infrared. The maximum CW output power of the DPSS lasers was >1 W, with an analog modulation BNC connector jack. The output power of the laser is proportional to the modulation voltage in 5 V range with maximum modulation frequency extending to >30 kHz. In this approach, an EG&G 7265 DSP lock-in amplifier was introduced for phase sensitive detection, the connections were quite simple using only the external jack of the instruments. The input of lock-in amplifier was connected to the detector output jack of the FTIR spectrometer, and its fast output jack was connected back to the external detector input jack A on the simultaneous synchronous technique module. The purpose of using the fast output port was to permit the pass through the signal at Fourier frequency and lock-in the PL signal at modulation frequency more effectively in DM mode, by selecting the time constant related to this port properly. The internal oscillator output of lock-in amplifier with sine wave output voltage adjustable from 0 to 5 V was connected to the laser driver to modulate the DPSS laser at expected lock-in frequency and to control the output power. Other option to modulate the DPSS laser was to use an external pulse generator (square wave was suitable); in this case, the lock-in amplifier was triggered by this pulse generator. By using this arrangement, in this approach the RS, DM, and SS modes could be software switchover freely without the changing of hardware and connections. This attachable PL and EL accessory was rather simple with only 4 optical parts; the operation was also quite easy. In this setup, a removable reflecting mirror...
could be simply inserted into the light path for EL measurements as shown in Fig. 1, in this case the trigger output from the EL driver was used as the reference for lock-in amplifier. As illustrated, this FTIR-PL accessory was for RT measurements without temperature control, the sample was simply put on the holder horizontally, the holder X-Y stage was useful to check the PL distribution across sample area. In the measurements, the operator just needed to put the sample on the focus point of the red He-Ne light from the emission port of FTIR spectrometer, and then steered and focused the DPSS laser light on the same point. In this setup, no further adjustment was needed when changing samples, therefore very suitable for daily examination of product. For low-temperature measurements, a similar accessory at a breadboard of the same size was also developed (not shown). Because in low-temperature measurements the samples were normally mounted on the holder of cryostat or Dewar vertically, a 2 in. reflecting mirror was added to turn 90° of the PL path for arranging the setup more conveniently. In case of using 1064 nm DPSS excitation laser, a small net video camera was attached for monitoring the laser beam at the computer screen.

### III. Demonstration and Discussion

To demonstrate the validity of the approach, a set of III–V or II–VI samples including substrates, bulk epitaxial layers, and device structures were measured using this setup. Those samples were chosen to cover a wider spectral span for the most interested NIR to MIR bands, and with a big difference in their PL intensities. Some details of the samples and FTIR spectrometer settings were listed in Table I. The samples are divided into two groups. The first group of sample, a to f, covers the spectral span of NIR-MIR from 11 500 to 2100 cm⁻¹ recommended for the combination of CaF₂ beamsplitter with InSb (77 K) detector, the second group of sample, g and h, covers the spectral span of MIR from 4000 to 600 cm⁻¹ recommended for the combination of KBr beamsplitter with MCT-A (77 K) detector. This work was not concentrated on the analysis of spectral details of the samples but the overall performance of the FTIR-PL system, so for easy comparison all the samples were measured at RT and at the same conditions for each group.

Figure 2 showed the measured PL of the first group of samples in RS mode, the BG signal (without sample) was also recorded. All the PL in Fig. 2 were recorded at a reasonable resolution of 16 cm⁻¹ with 32 scans at scan speed of 0.3165 cm/s, the data acquiring time for each PL was 25 s using this FTIR spectrometer. At this scan speed, the response time of the spectrometer was still comfortable to the operator, whereas at even lower scan speed the presetting window of the instrument became stagnant. In the measurements, a 532 nm DPSS excitation laser was used, its output power was fixed at about 0.3 W. From Fig. 2 it could be seen that, in RS mode, the shortwave side thermal BG arouses steeply from about 3500 cm⁻¹ for this quite sensitive InSb (77 K) detector. The strong CO₂ absorption around 2360 cm⁻¹ could be clearly seen. For samples a, b, and c with PL at NIR, normally the influence of BG is not a problem. Therefore, through the appropriate selection of the wavelength span even weaker PL could appear as far as it still exceeds the detection threshold of the FTIR. The zoom in of PL of sample b (a quaternary InAlGaAs layer) with quite weak intensity was also shown in Fig. 1, in this case the PL still could be seen clearly but with a low signal noise ratio (S/N) of only about 2.8. Further increase of the scan times has inconspicuous effects mainly due to the detector/amplifier noise. By using a more sensitive RT InGaAs detector, the S/N of sample b could be improved remarkably (not shown). For samples d, e, and f with PL entered MIR, different situations occurred. For samples d and e with rather strong PL compared to the corresponding thermal BG the PL still appears clearly, but if the PL is weak and submerged into BG the RS mode could not work properly. For sample f of an InAs substrate, its PL intensity was comparable to the BG. In this case, the PL just could be seen but had overlapped with BG. Subtracting the BG from the signal often showed poor results because of the random effects; in this case, the RS mode became inadequate. At even weaker PL intensity the RS mode was invalidated. For a sample with unknown features (peak wavelength, intensity, etc.) as in the case of most scientific research, collecting PL using RS mode in MIR is really a challenge.

Figure 3 showed the measured PL of the same group of samples in DM mode; the BG signal was also shown. All the PL were still recorded at 16 cm⁻¹ resolution with 32 scans at speed of 0.3165 cm/s, so the acquiring time for each PL kept unchanged for 25 s. In the measurements, the 532 nm DPSS excitation laser was modulated at about 35 kHz with average output power of about 0.3 W. In this wavelength span, the Fourier frequency was limited to about 7 kHz, so the time constant of lock-in amplifier was set to 80 μs (at slope of

<table>
<thead>
<tr>
<th>No.</th>
<th>Sample description</th>
<th>RT-PL peak (intensity)</th>
<th>FTIR setup</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>n-InP substrate</td>
<td>10 761 cm⁻¹ (28%)</td>
<td>11 500–2100 cm⁻¹</td>
</tr>
<tr>
<td>b</td>
<td>MBE n-In₀.52Al₀.22Ga₀.26As</td>
<td>8385 cm⁻¹ (0.68%)</td>
<td>InSb PD (77 K)</td>
</tr>
<tr>
<td>c</td>
<td>MBE n-In₀.53Ga₀.47As</td>
<td>5908 cm⁻¹ (89%)</td>
<td>CaF₂ beamsplitter</td>
</tr>
<tr>
<td>d</td>
<td>MBE n-In₀.84Ga₀.16As</td>
<td>3934 cm⁻¹ (36%)</td>
<td>R = 16 cm⁻¹, 32 scans</td>
</tr>
<tr>
<td>e</td>
<td>MBE p-In₀.85Al₁₁As/n-In₀.85Ga₁₁As</td>
<td>3518 cm⁻¹ (100%)</td>
<td>KBr beamsplitter</td>
</tr>
<tr>
<td>f</td>
<td>n-InAs substrate</td>
<td>2908 cm⁻¹ (44%)</td>
<td>R = 16 cm⁻¹, 32 scans</td>
</tr>
<tr>
<td>g</td>
<td>LPE HgCdTe</td>
<td>2130 cm⁻¹ (5.4%)</td>
<td>4000–600 cm⁻¹</td>
</tr>
<tr>
<td>h</td>
<td>MBE p-InSb/n-InSb</td>
<td>1590 cm⁻¹ (0.35%)</td>
<td>MCT-A PD (77 K)</td>
</tr>
</tbody>
</table>
6 dB/octave), a quite critical value for this span and speed. The sensitivity of lock-in amplifier was set to a high but unsaturated value of 5 mV. With those parameters the system still ran properly as demonstrated. From Fig. 3 it could be seen that, in DM mode the thermal BG has been suppressed effectively, all samples showed flat baselines. The PL signal of sample f inundated in the BG in RS mode emerged totally. The S/N in DM mode also improved obviously for the adoption of phase sensitive detection. For sample b with quite weak PL intensity the zoom in shows an increase of S/N from about 2.8 to 4.5, other samples with stronger PL intensities the S/N increased 3-4 times (for sample c from 62 to 200) experimentally. Notice that although from Figs. 2–5 all the y scales are in arbitrary units, their numerical value is quantitative for comparison. The signal intensity increased more than 2 orders in DM mode due to the introduction of lock-in amplifier; therefore detection threshold of FTIR (Ref. 16) was also improved.

For the detectors and lock-in amplifier the modulation frequency could be even higher (>100 kHz) facilely, whereas for this ordinary DPSS laser the modulation frequency was limited to <50 kHz experimentally. Therefore, if the spectral span extends to the shortwave side further, the scan speed still needs to slow down, say 2 times to 0.1571 cm/s. In this speed, the instrument behaved a little stagnant during presetting and data acquiring time increases somewhat, but all were still acceptable. At shortwave side, instead of thermal BG the stray light BG might also play an important role. Stray light BG in visible-NIR bands could be from sunlight or illumination, and blocking this BG totally is really a hard work. Also, staying in a full dark room is uncomfortable for the operator. At shortwave side, the stray light BG may form fake signal or even inundating PL signal in RS mode for the more sensitive detectors at this band (e.g., InGaAs or Si), whereas in DM or SS mode with modulated excitation source this unmodulated BG could also be removed effectively (not shown).

Figures 4 and 5 showed the measured PL of the second group samples g and h in RS and DM modes, respectively, at spectral span of 4000–600 cm$^{-1}$ of MIR band. The BG signals were also recorded. All the PL were still recorded at 16 cm$^{-1}$ resolution with 32 scans at speed of 0.3165 cm/s, the acquiring time for each PL is also 25 s. In the measurements, the 532 nm DPSS excitation laser was modulated at about 32 kHz with average power of about 0.3 W. In this wavelength span, the Fourier frequency was limited to about 2.5 kHz, the time constant of lock-in amplifier was still set to 80 μs. In this case, the second modulation frequency is >10 times of the Fourier frequency. The sensitivity of lock-in amplifier was set to a high but unsaturated value of 50 mV. Sample g was a liquid phase epitaxial grown HgCdTe bulk layer in mid-wave infrared band of 3–5 μm, sample h was a MBE grown InSb pn structure with PL at its bandgap wavelength of about
7.3 μm. Resolution of 16 cm\(^{-1}\) was just enough for most PL samples without fine spectral structure especially at RT. For a higher resolution of 8 cm\(^{-1}\), the acquiring time increased to 38 s correspondingly. Normally with the increase of wavelength in long wavelength side of MIR the PL intensity decreases dramatically, observation of PL at RT at longer wavelength becomes more difficult. From Fig. 4 it could be seen that, in RS mode with MCT-A detector at MIR band, the whole RT thermal BG appeared with peak around about 1000 cm\(^{-1}\). For samples g and h at RT, no noticeable PL signal could be found except the strong thermal BG. The recorded BG and “PL” curves were identical. Zoom in or subtracting the BG from the signal still resulted in no trace of the PL, implying that the PL of those samples were much weaker than the RT BG. By adopting DM mode, the PL signals were clearly extracted as shown in Fig. 5; the thermal BG was suppressed effectively. In this group of samples, the PL of sample g of a liquid phase epitaxial grown MCT material in mid-wave infrared was relatively “strong” with moderate S/N, in the curve a sharp absorption band of CO\(_2\) around 2360 cm\(^{-1}\) still occurred clearly. Through the purge of the FTIR spectrometer with dry nitrogen, those strong CO\(_2\) or H\(_2\)O related absorption bands could be weakened, whereas for this open-path FTIR-PL accessory they still could not be totally removed. Therefore, in case of those bands overlapped with useful PL information severely, a hermetic PL accessory box connected to the emission port of FTIR spectrometer should be developed air tight despite the inconvenience in operation. The PL intensity of sample h of a MBE grown InSb pn structure on GaAs substrate at RT was more than one order lower than sample g; however, in this case, the PL with some fine structures still could be differentiated clearly in DM mode but with lower S/N. Notice that for those type of samples in MIR the PL were only observed at low temperatures;\(^{1,6-12}\) therefore, the overall performance of DM mode using DPSS excitation laser modulated at appropriate frequency was favorable. Observing the PL of sample h and BG data in DM mode, a very weak residual BG hill (seems around the position of thermal BG) with its amplitude comparable to the noise floor could be noticed. We find that this noise hill was related to the electromagnetic interference between the accessory lock-in amplifier/laser driver and FTIR spectrometer. Through appropriate grounding this interference had been weakened effectively. In experiments, it is also noticed that, due to the introduction of second modulation frequency, some extra interference or noise at unpredictable wavenumber zone might be presented. Those interference or noise was mostly possibly caused by the weaker cross modulation components of the second modulation frequency with the Fourier signal frequency in the lock-in amplifier though it was set out off the Fourier frequency far away.

Through the fine adjustment of the excitation laser modulation frequency, those interference or noise could be removed or suppressed adequately. In experiments, the DPSS excitation laser modulation frequency was adjusted finely around the setting value to see the cleanness (free of interference peak or strong noise, without sample) of the monitor wavenumber window in presenting mode of the FTIR spectrometer, or the wavelength span was shifted/narrowed to keeping it away from interference peak or strong noise zone. In this case, the better S/N could be reached for the acquired data.

In Fig. 5, the PL of sample h in SS mode was also shown, these data were acquired still at resolution of 16 cm\(^{-1}\) but only in one scan. The DPSS laser was still modulated at the same frequency. From this trace it could be seen that, even using only one scan the S/N is better that in DM mode. In SS mode, adopting this approach the excitation laser modulation frequency is more appropriate for the lock-in amplifier to reach lower noise, also the Fourier frequency is “zero” so have no possible cross interference with the excitation laser modulation frequency, and therefore a better S/N was achieved even at only one scan. However, the data acquiring time for one scan in SS mode at the same spectral span was 7.4 min, a still acceptable but much longer value than those of 32 scans in RS or DM modes. For 32 scans in SS mode, data acquiring time was increased to an unpractical value of 3.8 h. Table II compared the data acquiring time of each mode at certain conditions.

<table>
<thead>
<tr>
<th>TABLE II. Comparison of the data acquiring time of each mode at certain conditions.</th>
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<tbody>
<tr>
<td><strong>4000–600 cm(^{-1})</strong></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>1 scan</td>
</tr>
<tr>
<td>32 scans</td>
</tr>
</tbody>
</table>

In SS mode, adopting this approach the excitation laser modulation frequency was adjusted finely around the setting value to see the cleanness (free of interference peak or strong noise, without sample) of the monitor wavenumber window in presenting mode of the FTIR spectrometer, or the wavelength span was shifted/narrowed to keeping it away from interference peak or strong noise zone. In this case, the better S/N could be reached for the acquired data.
In SS mode, the highest resolution is normally lower than those in RS mode (0.5 cm$^{-1}$ in this spectrometer) because of the oppressive data acquiring time at high resolution and servo precision of the spectrometer. If the highest resolution in SS mode was still enough for device characterization, the spectral span should be as narrow as possible for decreasing the total data acquiring time, in this case the SS mode still could be a useful spare mode.

As a complementary, EL spectra of a quantum cascade laser at room temperature around 7.7 μm were also measured in RS, DM, and SS modes, respectively, at the same driving condition, as shown in Fig. 6. This sample was driven electrically below its threshold using pulses of 200 ns duration at repeat frequency of 50 kHz. The cavity length was 3 mm. The EL intensity is normally much weaker than the lasing intensity; at MIR band it is often comparable to the thermal BG. As shown in the left side of Fig. 6, in RS mode at the highest resolution of 0.125 cm$^{-1}$ of this instrument with one scan, the EL signal could be seen clearly but overlapped on the thermal BG, some absorption features of the water vapor on the thermal BG also appeared. The acquiring time for this spectrum was 15 s at scan speed of 0.9495 cm/s. In this case, the resolution was almost enough but the BG is really too high, therefore the data were unsuitable for extracting the gain/loss features of the cavity using Hakki-Paoli$^{18}$ or modified methods. The right side of Fig. 6 showed the spectra in DM mode at the same acquiring time, the thermal BG was totally restrained and the data quality improved remarkably. This data with appropriate contrast as shown in the inset of Fig. 6 was good enough for extracting the gain/loss features. The EL spectrum was also measured using SS mode at 32 times lower resolution of 4 cm$^{-1}$ with one scan as also shown in the right side, in this case the S/N was good but the acquiring time increased dramatically to 1268 s at the same wavelength span. Notice that the highest resolution in SS mode was limited to a lower value of 0.5 cm$^{-1}$ in this instrument for the oppressive long acquiring time (>2.5 h at 0.5 cm$^{-1}$ resolution, 100 cm$^{-1}$ wavelength span with one scan), the chirp of the sample and random effect during this long acquiring time were uncontrollable, therefore the SS mode was unsuitable for acquiring fine spectral features.

![Figure 6](image.png)

**FIG. 6.** Measured EL spectra of a room temperature quantum cascade laser around 7.7 μm in RS, DM, and SS modes. The inset was the zoom in of the spectrum in DM mode.

### IV. CONCLUSION

In conclusion, an improved FTIR spectroscopy approach for measurements of PL and EL in mid-infrared has been developed, in which DPSS laser was adopted as PL excitation source. In this approach, three different modes of RS, DM, and SS were software switchable without changing of the hardware or connections. The advantage and drawback of each mode were discussed in detail. Using this approach a group of samples extending from NIR to MIR with quite different PL intensities have been characterized to demonstrate the validity of the system. The weaker electroluminescence of quantum cascade lasers in mid-infrared band was also surveyed at different resolutions. Results show that for samples with relatively strong PL or EL out off the BG, RS mode is preferable. For weaker PL overlapped with BG, DM is the most effective mode. To get a better S/N when weaker PL or EL signal has been observed in DM mode, SS mode should be an advisable option despite its long data acquiring time and limited resolution.

### ACKNOWLEDGMENTS

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